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2014 J. Phys.: Conf. Ser. 507 022044

(http://iopscience.iop.org/1742-6596/507/2/022044)

Effect of heat treatment on superconducting properties of FeSe wire fabricated by PIT method

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Abstract.

We report on successful fabrication of superconducting FeSe wire using hot gas extrusion (HGE) ex-situ PIT (powder in tube) method. Length of the obtained wire was about 60cm with cross-sectional superconducting core area nearly 2.5×10^{-3} cm^2. For the wire sample we observed superconducting transition temperature, $T_c^{onset} \approx 11$ K, about 1.2K lower compared to preliminary prepared FeSe powder. Heat treatment in argon atmosphere at 350^{o}C resulted in transition width decrease from $\triangle T(10\% - 90\%) \approx 1.75$ K in sample without heat treatment down to $\triangle T \approx 0.9$ K in annealed samples. Estimated derivative of the upper critical field as a function of temperature of the sample annealed during 72h in argon atmosphere at 350^{o}C is $dH_{c2}/dT \sim -2.9$ T/K. Applying WHH theory to our data allows to define $H_{c2}(0K) = 0.69T_c \times (dH_{c2}/dT) \approx 19.8T$. The untreated wire shows critical current density, $J_c \sim 75$ A/cm^2 at T=4.0 K in zero field. Increasing annealing time up to 72 hours at 350^{o}C in argon atmosphere gives rise to J_c increase of about 60% approaching 120 A/cm^2 at T=4.0K and H=0T . Also J_c measurements were made in magnetic fields up to 9T. Our results show applicability of the HGE PIT method for fabrication of superconducting wires based on FeSe compound. Long-range heat treatment is necessary to improve superconducting properties of the samples.

1. Introduction

Recently discovered iron-based superconductors have superconducting critical temperature (T_c) approaching 55K [1] that is below liquid nitrogen temperature in contrast to high-T_c cuprate superconductors (HTSC) with T_c up to about 130K. However, already earliest studies of iron-based superconductors have shown great potential of these compounds for high-current applications in high magnetic fields. Obvious advantages of iron-based superconductors are the very high upper critical field (H_{c2}) of about 150-200T [2–4], low anisotropy in comparison with HTSC, and high values of the critical current density (J_c), exceeding 10^6 A/cm^2 at 4.2K in zero field [5–8]. FeSe is the simplest iron-containing superconductor from the point of view of its chemical composition. It has tetragonal structure formed by alternating iron and selenium layers [9, 10]. T_c of FeSe compound is near 12K at normal conditions [11, 12] and increases up to 37K under external pressure of about 5 GPa [13]. FeSe may be easily synthesized compared to other families of iron-based superconductors and, furthermore, does not contain toxic arsenic.

For this reason FeSe may be considered as the model iron-based superconductor to check the possibility to produce superconducting wires and tapes using various methods. Experiments with this compound are useful to get necessary technical experience and also could help to understand the main factors influencing superconducting properties of wires and tapes made using iron-based superconductors. The basic requirement to a quality of the superconducting wire is T_c, J_c, and H_{c2} values close to values of these parameters obtained in bulk samples. The main technique of manufacturing of superconducting wire is the PIT (powder in the tube) method. This method has two different modifications known as in-situ and ex-situ [14]. The first method suggests producing of the superconducting wire from the mixture of initial components, when superconductive phase is obtained inside the wire as a result of heat treatment. The second method requires preparing of preliminary synthesized polycrystalline superconductor. To date, there are already some reports concerning manufacturing of superconducting wires and tapes using FeSe [9, 12, 15]. The highest J_c values up to 10^3 A/cm^2, in 7-core wire were obtained using gas diffusion technique [15]. About two times lower J_c values were reported for 3-core wire produced by chemical transformation PIT process [9].

In this work the single-core superconducting wire on the basis of FeSe was made by ex-situ HGE method. We report on production of the FeSe superconducting wire and its transport properties in fields up to 14T. Our results indicate that the critical current density J_c approaches 120 A/cm^2(T=4.0K, H=0T) after low temperature (350^{o}C) heat treatment in argon during 72h.

2. Experiment and discussion.

The FeSe wire was prepared using the ex-situ HGE PIT technique with steal C22(DIN) sheath in several intermediate steps. Firstly, stoichiometrical mixture of Se and Fe pieces was sealed into quartz tube and synthesized during 120h at temperature near 900^{o}C. The phase composition of the synthesized polycrystalline FeSe was defined by X-ray powder diffraction method. The results of X-ray studies showed that the tetragonal FeSe is a main phase with amount above 90%. Then superconductive material was regrounded, packed into a steel tube of 9.3 mm outer diameter and 3 mm wall thickness and plugged as shown in Fig.1a and Fig.1b by a steel fitting. After packing, the tube with FeSe powder was drawn to wires of 2.2 mm in diameter and 600mm in lengths using HGE method (Fig.1c and Fig.1d) at argon pressure of about 330-340 MPa and temperature of about 950^{o}C. Prepared wire (Fig. 2a) was cut into 1 cm lengths specimens (Fig. 2b), and finally, a few batches of these samples were subjected to heat treatment in argon atmosphere at 350^{o}C during 24, 48, 72, and 96 hours. Fig. 3 shows temperature dependence of zero field resistance of polycrystalline samples without heat treatment and annealed at 350^{o}C during 24, 48, 72, and 96 hours. One can see that the superconducting transition of preliminary prepared polycrystalline sample starts at $T_c^{onset} \approx 12.1K$ with transition width (10%-90%) of about $\triangle T \approx 3^o$C . This value is similar to data obtained in other studies [10, 11]. For our wire samples superconducting transition starts at lower temperature while the transition width gradually decreased to the value below 1K. It should be also noted that with increasing temperature resistive response for all samples with heat treatment up to 72 hours appears at the same temperature $\sim$ 9.5K indicating stability of FeSe phase in our wire samples. Higher heat treatment time resulted in displacement of the resistivity onset to lower temperatures.

Further, we measured temperature dependence of the resistance of our wire samples near superconducting transition in magnetic fields up to 14T (up to 9T using PPMS system and up to 14T with Oxford magnetic system). As an example in Fig.4 we show resistive superconductive transitions of FeSe wire sample annealed during 24h with probing current of 5 mA. Similar results without any systematic dependence on annealing time were obtained for the samples subjected to the heat treatment up to 96 hours at 350^{o}C in argon atmosphere. These measurements clearly show presence of superconductivity in our wire samples in magnetic fields up to 12T at liquid helium temperature 4.2K. From the linear extrapolation of the $H_{c2}(T)$ dependence

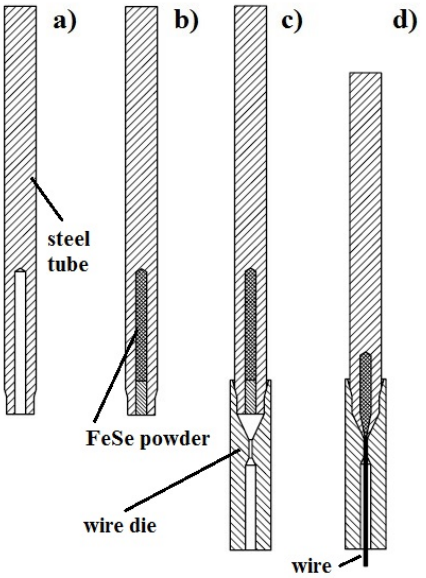

Figure 1. (a) original steel tube; (b) tube with FeSe powder; (c) setup for HGE; (d) HGE process.

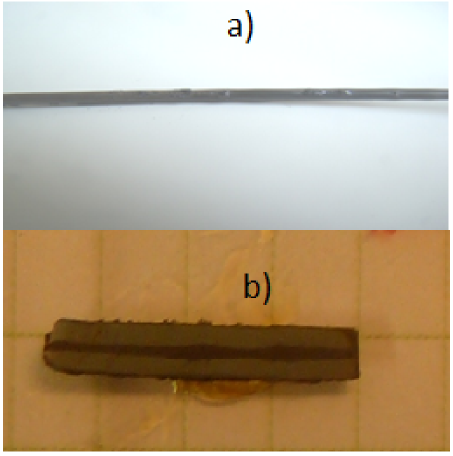

Figure 2. (a) superconducting FeSe wire of 2.2 mm diameter obtained by gas extrusion method; (b) longitudinal cross-section of the FeSe wire.

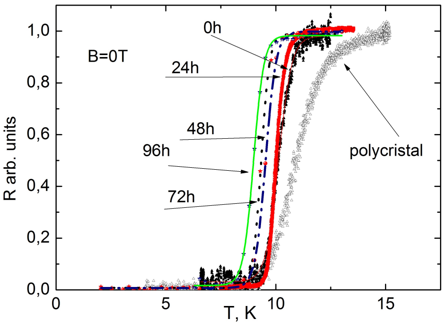

Figure 3. Zero field R(T) dependence for wire FeSe samples with different heat treatment in the region of superconducting transition.

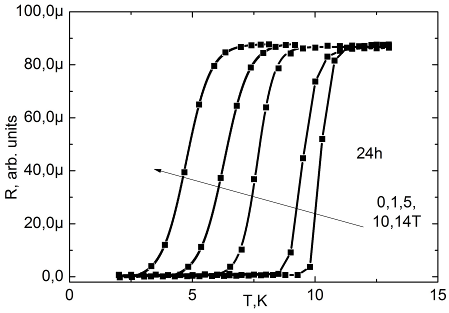

Figure 4. Temperature dependence of resistance below 14K for the as-prepared FeSe wire under magnetic fields up to 14 T.

constructed using mid-point of the resistive superconducting transition $H^*_{c2}(0)$ value nearly 31T may be obtained. Applying the Werthamer, Helfand, Hohenberg (WHH) expression $H_{c2}(0K) = 0.69T_c \times (dH_{c2}/dT)$ [16] we get the value of the upper critical field $H_{c2}(0) = 19.8T$ for our FeSe wire samples.

Transport critical current (I_c) was measured on 1 cm length samples by a standard four-probe resistive method at different temperatures below T_c and applied magnetic fields up to 9T using a Physical Property Measurement System (PPMS). The magnetic field was applied perpendicularly to the wire axis. I_c value was defined with 1 μV/cm criterion. To get J_c, I_c values were normalized to the smallest cross-sectional area of 1 cm sample found after inspection under optical microscope of the cross-section of the wire along its length. In Fig. 5 and Fig. 6 we show $J_c(T)$ dependence, for obtained in our experiment FeSe wires in zero field and 9T field. One can clearly see essential increase of J_c for samples with increasing heat treatment time.

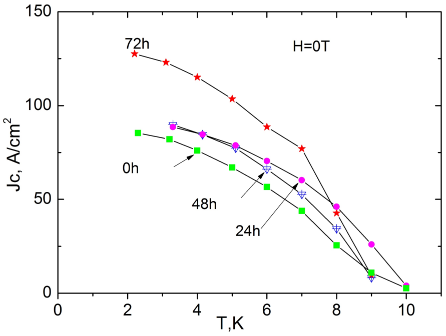

Figure 5. Temperature dependence of critical current density for the obtained single core FeSe wires at zero applied magnetic field.

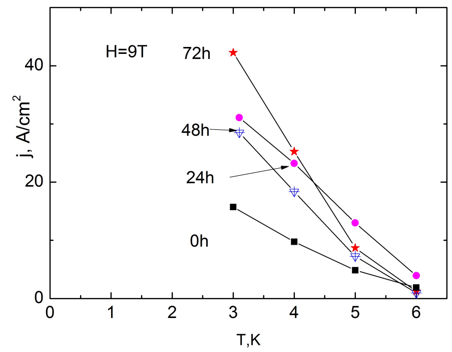

Figure 6. Temperature dependence of critical current density for the obtained single core FeSe wires in magnetic field 9T

In particular, enhancement of J_c about 60% after 72h annealing (T=4.0K, H=0T) indicates important role of heat treatment at 350^{o}C in argon atmosphere to increase critical current density of FeSe wires prepared using HGE method. Our data are below results reported by other groups [9, 12, 15]. The probable reason is porous microstructure of our wires observed under high resolution optical microscope.

In conclusion, in our study we show that PIT technique with hot gas extrusion procedure allows to produce FeSe wires with reasonable superconducting parameters. Long time (up to 72 hours) heat treatment at 350^{o}C in argon atmosphere results in essential increase of J_c of FeSe wire prepared by this method.

3. Acknowledgements

This study was supported by RFBR (grant N 13-02-01180) and by grants of Ministry of Education and Science of the Russian Federation.

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